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Patentanmeldung Nr. Patent application No. Demande de brevet n°

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R C van Dijk

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SUEDE

Bezeichnung der Erfindung/Title of the invention/Titre de l'invention:
(Falls die Bezeichnung der Erfindung nicht angegeben ist, siehe Beschreibung.
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METHOD AND APPARATUS FOR CONTROL OF CHEMICAL REACTIONS

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METHOD AND APPARATUS FOR CONTROL OF CHEMICAL REACTIONS

Technical Field

This invention relates to a method and apparatus for performing microwave assisted chemical reactions, in particular for performing microwave assisted organic synthesis reactions under nearly ideal heating and cooling conditions.

Background Art

In the field of chemical reactions and particularly of chemical synthesis, there is a high desirability to prepare products with a high purity and yield. Thus, usually each chemical reaction has to be performed under optimum reaction conditions that promote formation of desired reaction product and prevent unwanted side products and/or degradation of the desired final product. Occurrence of side reactions and degradation of the desired product usually takes place during the heating and cooling phase of a chemical reaction, depending on, inter alia, the heating respectively the cooling rate and how uniform the reaction mixture is heated respectively cooled. Too slow heating or cooling rate and/or non-uniform heating or cooling of the reaction mixture often results in products with a lower degree of purity and yield.

Furthermore, it is known that microwave heating enables almost instantaneous heating of materials in a fast and uniform manner. The rapid uniform heating provided by microwave heating is offering higher purity of the resulting product depending, inter alia, that impurities due to side reactions are diminished. However, cooling of the reaction products is still performed using conventional thermal conduction cooling means, e.g., heat exchangers. These conventional cooling means are slow and non-uniform and thus, final products with a lower degree of purity and yield are obtained due to, e.g., degradation of the formed product or side reactions. Accordingly, the gained higher purity and yield of the desired product during the microwave heating step cannot be maintained, but will actually lessen during the cooling step for the reasons set out hereinabove.

Therefore, there is a strong need for a way of obtaining a method and apparatus for performing chemical reactions, where microwave heating is combined with a rapid, uniform cooling, so that both the heating and cooling step take place instantaneously and uniformly, resulting in final products with improved yield and purity.

US-5,932.075 relates to an apparatus for performing batch-wise chemical reactions using microwave energy in a vessel wherein cooling is carried out by means of a heat exchanger means (cold finger) immersed in the contents of the vessel under high temperature and pressure conditions.

US-5,287,397 relates to a method of performing chemical reactions on a continuous basis using microwave heating and cooling the reaction by using a heat exchanger means .

Both US-5,932.075 and US-5,287,397 use traditional heat exchanger cooling means where the cooling rate is much slower than the heating rate achieved by microwave heating and thereby the product yield and purity after the heating step is decreasing during the cooling step.

The object of the present invention is to provide a method and apparatus that solve the above-mentioned problems.

Summary of the Invention

The above-mentioned object is achieved by the present invention as defined in the independent claims. Preferred embodiments are set forth in the dependent claims.

An ideal chemical reaction should be heated so that the desired temperature is reached instantaneously and uniformly, and then kept constant for a desired time in order to carry out the reaction to maximum yield is obtained. Subsequently, it should be cooled so that the desired lower temperature is reached instantaneously and uniformly. In order to perform a chemical reaction as close as possible to the ideal heating and cooling profile, the reaction mixture should be heated respectively cooled almost instantaneously and uniformly, to the desired temperature.

Adiabatic cooling as defined herein means the cooling of the contents of a reaction chamber that takes place instantaneously and uniformly; so that no heat (energy) enters or leaves the system and no portion of the contents of the reaction chamber leaves the system (being lost into the surroundings).

The method and the apparatus according to the claimed invention are providing for chemical reactions resulting in products with improved yield and purity.

The method according to the present invention comprises:

- supplying substances for a chemical reaction into a reaction chamber, which is adapted to withstand high temperature and pressure ,
- applying microwave heating to initiate the chemical reaction and reach a desired temperature,
- cooling the reaction mixture to a desired lower temperature by using adiabatic cooling.

The apparatus according to the present invention comprises a reaction chamber having at least an inlet and an outlet and adapted to withstand high temperature and pressure, a microwave heating source for heating a reaction mixture and a cooling means for adiabatic cooling of the reaction mixture.

Brief Description Of The Drawings

Figure 1 shows a heating/cooling profile obtained by conventional heating and cooling methods and apparatuses (dashed line) and an ideal heating/cooling profile which is aimed at by means of the method and apparatus according to the present invention (unbroken line)

Figure 2 shows a preferred embodiment according to the present invention illustrating a reaction chamber with a movable wall,

Figure 3 shows another preferred embodiment according to the present invention illustrating a reaction chamber operationally connected to an expansion vessel.

Detailed Description Of The Invention

The present invention will now become more fully understood from the detailed description given herein, wherein reference is made to the accompanying drawings.

In the following description of embodiments of the present invention, similar elements in different embodiments are denoted with the same reference numerals.

In Fig. 1, an ideal heating and cooling profile 1 respectively a conventional one 2 for a chemical reaction, are illustrated. A chemical reaction performed according to the ideal heating and cooling profile will be heated from an initial temperature T_i to a desired higher temperature T_d, instantaneously and uniformly, then if desired be kept for any desired time at T_d, and finally cooled to a desired lower temperature T_c, also instantaneously and uniformly. Heating and/or cooling of a chemical reaction, taking place within the area 3 (the area between the conventional 2, respectively the ideal 1 heating and cooling profile) will result in final products with a lower purity and yield.

The equipment used for performing microwave heated chemical reactions, usually includes a device having a cavity into which microwaves are guided from a microwave source, typically a magnetron. Such equipment is well known to those skilled in the art and is not, therefore, described in detail herein.

Adiabatic cooling according to the present invention may be obtained by changing the volume of the reaction mixture. This may be achieved either by varying the volume of the reaction chamber or by letting the reaction mixture expand into another vessel operationally connected with the reaction chamber.

A preferred embodiment of the apparatus according to the present invention is shown in Figure 2. In this embodiment, the volume of the reaction chamber 4 is variable by means of a moveable wall 5, e.g, in the form of a piston. As the piston 5 changes its position in the reaction chamber 4, the reaction mixture contained in the

reaction chamber will instantaneously change its temperature in accordance with the change in volume of the reaction chamber 4.

A further preferred embodiment of the inventión is shown in Figure 3. In this embodiment, the reaction chamber 4 is operationally connected with an expansion vessel 9 via a valve 8, in the way that is shown in the figure. The reaction chamber, having a volume V_1, is partly filled with a reaction mixture. The expansion vessel has a volume V_0, which is larger than that of the reaction chamber 4 and, preferably, ambient pressure P_0 and temperature T_0, when valve 8 is closed. While the reaction mixture in the reaction chamber 4 is heated to a temparature T_1 and the pressure is increased to P_1, valve 8 is closed. The reaction mixture in the reaction chamber 4 comprises two phases, a liquid and/or solid phase 7 and a vapor phase 6 due to the prevailing temperature and pressure conditions. When cooling is needed, the valve is opened, causing any liquid or slurry or solid particles at the lower end of the reaction chamber 4, to flow into the expansion vessel 9. The pressure difference, between the reaction chamber 4 and the expansion vessel, forces the reaction mixture to flow into the expansion vessel. During this process the whole reaction mixture is loosing heat under adiabatic cooling conditions. Thermal conduction of the reaction mixture on the walls of the expansion vessel 9 will also contribute to some extent to lower the temperature of the reaction mixture. If the expansion vessel 9 has a volume Vo that is sufficiently larger than the volume V_1 of the reaction chamber 4 the final temparture and pressure in the expansion vessel 9, T_2 and P_2 respectively, will be slightly higher than that of the initial T_0 and P_0 respectively.

The achieved cooling rate will depend on the reaction volume, reagents, solvents and reactants used and temperature conditions, but will be significantly faster than using conventional cooling techniques. As an example 200 ml of water has been shown to cool from 200 °C to 40 °C in seconds rather than in minutes or hours as is the case with conventional cooling means.

The pressure used in the present invention may be up to 1000 bar. The temperature used may be up to 500 °C. The time intervals used for the cooling process may vary from parts of seconds to a few minutes.

The apparatus according to the present invention may also comprise control means for control various parameters that are important, such as microwave input power, pressure, temperature, etc.. in order, for example, to obtain the desired temperature profile for a certain chemical reaction. These control means may be any known control means suitable for use with the present invention.

The invention provides for chemical products with improved purity and thereby simplifies the subsequent purification process.

The method and the apparatus according to the present invention can be utilised for performing chemical reactions on a batch as well as on a continuous basis.

The method and the apparatus according to the present invention are suitable for performing chemical reactions and particularly chemical synthesis reactions, in laboratory scale as well as in large industrial scale. They are especially suitable for performing chemical reactions in large scale.

Furthermore, the method and apparatus of the invention is particularly suitable for organic synthesis reactions and is especially suitable for the production of labile molecules.

The present invention also relates to the use of the above-described method and apparatus for performing organic chemical synthesis reactions. Chemical reactions that can be carried out by using the hereinabove described method and apparatus are, for example, oxidation, nucleophilic substitution, addition, esterification, transesterification, acetalisation, transketalisation, amidation, hydrolyses, isomerisation, condensation, decarboxylation and elimination.

Claims

1. A method of performing chemical reactions comprising:
- supplying substances for a chemical reaction into a reaction chamber(4), which is adapted to withstand high temperature and pressure ,
- applying microwave heating to initiate the chemical reaction and reach a desired temperature and,
- cooling the reaction mixture to a desired lower temperature by using adiabatic cooling.

2. A method according to claim 1, further comprising
- maintaining the desired temperature substantially constant for a desired time to carry out the chemical reaction.

3. A method according to claim 1 or 2, wherein the cooling is obtained by increasing the volume of the reaction chamber (4) by moving a moveable wall that is a part of the reaction chamber.

4. A method according to claim 1 or 2, wherein the cooling is obtained by increasing the volume of the reaction mixture by letting it expand into an expansion vessel.

5. An apparatus for performing the method according to any one of claims 1-4, comprising a reaction chamber (4) having at least an inlet and an outlet and adapted to withstand high temperature and pressure, a microwave heating source for heating a reaction mixture and a cooling means for adiabatic cooling of the reaction mixture.

6. Apparatus according to claim 5, wherein the reaction chamber (4) comprises a moveable wall (5).

7. Apparatus according to claim 5, wherein it further comprises one or more expansion vessels(9) operationally connected to the reaction chamber (4) through an outlet in the reaction chamber (4).

8. Apparatus according to claim 7, wherein the outlet is positioned at the lower end of the reaction chamber (4) at or below the liquid phase level of the reaction mixture.

9. Use of a method according to any of claims 1-4 for performing organic synthesis reactions.

10. Use of an apparatus according to claims 5-8 for performing organic synthesis reactions.

Abstract

This invention relates to a method and apparatus for performing microwave assisted chemical reactions, in particular for performing microwave assisted organic synthesis reactions under nearly ideal heating and cooling conditions.

The method according to the present invention comprises, supplying substances for a chemical reaction into a reaction chamber, which is adapted to withstand high temperature and pressure , applying microwave heating to initiate the chemical reaction and reach a desired temperature, and cooling the reaction mixture to a desired lower emperature by using adiabatic cooling. The invention also relates to an apparatus for performing the method and use of the method and apparatus for performing organic synthesis reactions.

Fig. 1

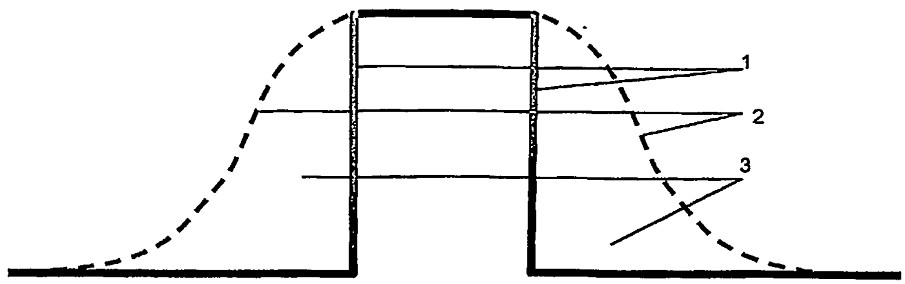


Fig. 2

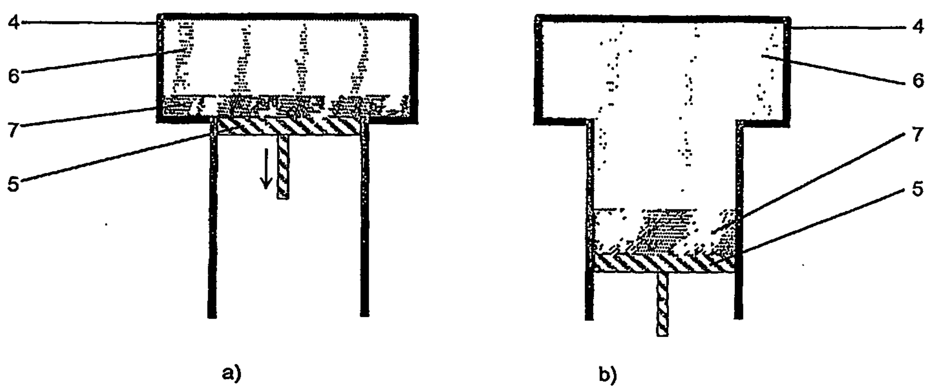
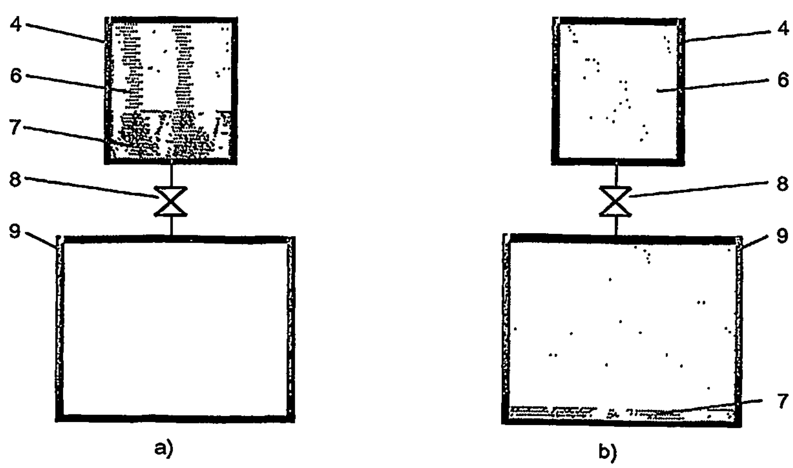


Fig. 3



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